

LA-UR-17-28474

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Title: Isotopic Composition Measurements Using Ratio Based Method

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Intended for: Training course

Issued: 2017-09-19

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**NONPROLIFERATION AND
ARMS CONTROL (NPAC)**

Isotopic Composition Measurements using Ratio Based Method

Fundamentals of Non-Destructive Assay for International Safeguards

Los Alamos National Laboratory

September 26, 2017

Duc Vo

Los Alamos National Laboratory

-  **SAFEGUARD** NUCLEAR MATERIALS TO PREVENT THEIR DIVERSION OR THEFT
-  **CONTROL** THE SPREAD OF WMD-RELATED MATERIAL, EQUIPMENT AND TECHNOLOGY
-  **NEGOTIATE, MONITOR AND VERIFY** COMPLIANCE WITH INTERNATIONAL NONPROLIFERATION AND ARMS CONTROL TREATIES AND AGREEMENTS
-  **DEVELOP** PROGRAMS AND STRATEGIES TO ADDRESS EMERGING NONPROLIFERATION AND ARMS CONTROL THREATS AND CHALLENGES

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Estimated Module Duration: 40 minutes

Required Tools and Materials:

1. Projector, screen, laptop with Word and PowerPoint programs
2. Participant guides, with slides and supplemental material

References:

1. Passive Nondestructive Assay of Nuclear Materials (PANDA):
<http://www.lanl.gov/orgs/n/n1/panda/>
2. FRAM Manual (included in C:\FRAMData\References)

Supporting Documents:

1. None

Job Aids:

1. None

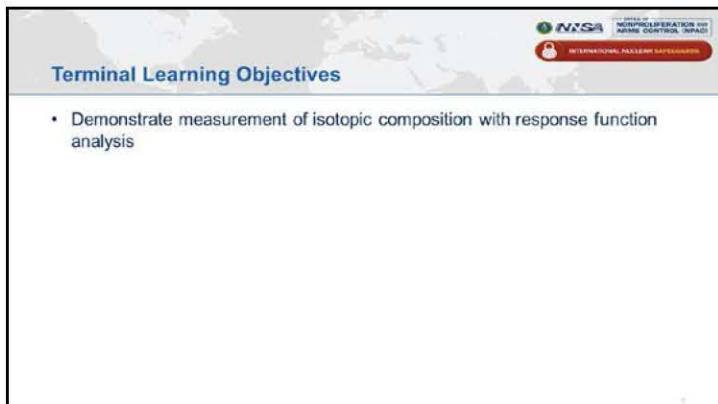
Terminal Learning Objectives (TLOs):

- TLO-1: Demonstrate measurement of isotopic composition with response function analysis

Enabling Learning Objectives (ELOs):

- ELO-1: State the categories of uranium and plutonium
- ELO-2: Discuss basic principles of response function analysis
- ELO-3: Describe the relative efficiency determination
- ELO-4: List conditions required for response function analysis
- ELO-5: Measure plutonium and uranium isotopic composition

Additional Information for Students:



Terminal Learning Objectives


- Demonstrate measurement of isotopic composition with response function analysis


This lecture is focused on the isotopic ratio method. In the lab, we will use two different analysis programs: MGA/MGAU (developed by LLNL) and FRAM (developed by LANL).

Instructor Notes:

Review learning objectives with the participants.
 Encourage participants to ask questions during the lecture.

Additional Information for Students:





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Enabling Learning Objectives

- State the categories of uranium and plutonium
- Discuss basic principles of response function analysis
- Describe the relative efficiency determination
- List conditions required for response function analysis
- Measure plutonium and uranium isotopic composition

Instructor Notes:

Review learning objectives with the participants.
 Encourage participants to ask questions during the lecture.

Additional Information for Students:



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Categories of Uranium

- **Only three isotopes of uranium are found in nature:**
 ^{238}U (99.27%), ^{235}U (0.720%), ^{234}U (0.006%)
- **Categories of Enrichment (E = % of ^{235}U)**
 - Depleted Uranium (DU) E < 0.72 %
 - Natural Uranium (NU) E = 0.72 %
 - Enriched Uranium E > 0.72%
 - Low Enriched Uranium (LEU) 0.72% < E < 20.0 %
 - High Enriched Uranium (HEU) E ≥ 20.0 %

Instructor Notes:

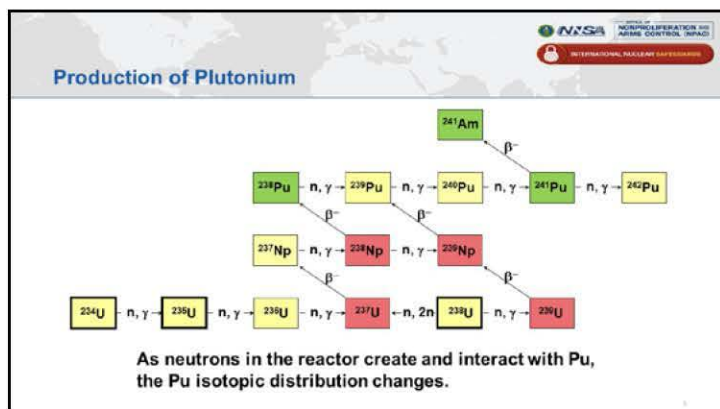
Natural uranium constitutes of three naturally occurring isotopes: ^{238}U , ^{235}U and ^{234}U . Their Natural Abundance (NA), referred as the abundance of isotopes of a chemical element as naturally found on a planet, is respectively, 99.27%, 0.720% and 0.006%. ^{238}U is the most abundant and has the longest lifetime (4.5 billion years). It is not very radioactive and neutron capture by this nucleus leads to the formation of fissile ^{239}Pu in a reactor.

^{235}U is the only fissile nucleus found in natural uranium. ^{234}U is found in a very small percentage and occurs as an indirect decay product of ^{238}U .

Depleted uranium has a lower content of ^{235}U (<0.72%) than natural uranium. Most DU comes as a by-product of the production of enriched uranium for use as fuel in nuclear reactors and in the manufacture of nuclear weapons.

Enriched uranium (EU) is used in nuclear reactors that need a higher concentration (enrichment) of ^{235}U (>0.72%) than which exists in natural uranium. There are two types of enrichment: Low Enriched Uranium (LEU) (most commercial reactor fuel is enriched to 5%) and Highly Enriched Uranium (HEU).

Additional Information for Students:



Plutonium does not exist naturally. Instead, it is created in reactors. Pu-239 is a decay product of Np-239, which is a decay product of U-239, which is created when a U-238 atom absorbs a neutron. Pu-240 is created whenever Pu-239 absorbs a neutron. Thus, high burnup fuel will have more Pu-240 relative to Pu-239 than low burnup fuel (see next slide).

Instructor Notes:

All plutonium originates in nuclear reactors and is produced by capturing extra neutrons by ^{238}U to form ^{239}U , which then undergoes a series of decays to form ^{239}Pu . Plutonium does not exist in nature but if you take ^{238}U , which makes up the overwhelming fraction of natural uranium and bombard it with neutrons, some nuclei will absorb a neutron, transforming them into ^{239}U . This nucleus has too many neutrons to be stable, and decays by beta decay transforming one of the neutrons in the nucleus to a proton, which transmutes the ^{239}U into Neptunium, ^{239}Np . ^{239}Np , while more stable than ^{239}U , remains unstable and undergoes beta decay, resulting in ^{239}Pu which can be considered as stable. The isotopic composition of plutonium is affected by how long it stays in the reactor. Short exposure produce plutonium with very little ^{240}Pu and with very little plutonium being consumed by fission. Long exposure produce high ^{240}Pu concentrations, and a substantial portion of the plutonium produced is consumed by fission.

Additional Information for Students:

Categories Plutonium				
	Low Burnup → High Burnup			
^{238}Pu	0.02	0.3	0.8	1.6
^{239}Pu	93.5	82.5	73.3	57.3
^{240}Pu	6.2	13.8	18.3	25.0
$^{241}\text{Pu} + ^{241}\text{Am}$	0.3	3.8	6.6	11.7
^{242}Pu	0.04	0.8	2.1	5.6


IAEA Definitions:

- Weapons-Grade <7% ^{240}Pu or >93% ^{239}Pu
- Reactor-Grade >7% ^{240}Pu or <93% ^{239}Pu


Weapons-grade plutonium (WGPu) can be extracted from low burnup fuel.

Instructor Notes:

Additional Information for Students:



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Neutron Coincidence Counting

Determination of mass requires the isotopic composition measurements to be combined with another measurement. Typically a weight or neutron measurement. For example:

- Combine a weight with enrichment measurement to get ^{235}U mass
- Combine a neutron $^{240}\text{Pu}_{\text{eff}}$ mass with isotopic composition to get total Pu mass


$$^{240}\text{Pu}_{\text{eff}} = 2.52 \times \frac{^{238}\text{Pu} + 240\text{Pu} + 1.68 \times 242\text{Pu}}{m_{^{240}\text{Pu}_{\text{eff}}} \text{ (from neutron counting)}}$$

$$m_T = \frac{^{240}\text{Pu}_{\text{eff}} \text{ (from isotopics)}}{m_{^{240}\text{Pu}_{\text{eff}}} \text{ (from neutron counting)}}$$

Instructor Notes:

Nuclear material assay aims to determine the mass of each isotope as an item, typically by combining gamma spectroscopy with neutron coincidence counting. All three of the even isotopes contribute to the response of a neutron-coincidence counter. The contribution from ^{240}Pu dominates for most plutonium bearing materials. Therefore the ^{240}Pu mass is defined by the equation, where $^{240}\text{Pu}_{\text{eff}}$ is the mass of ^{240}Pu that would give the same coincidence response as that observed from the actual measured item.

Additional Information for Students:



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Important Isotopes for Plutonium Mass

Typical plutonium consists of almost all ^{239}Pu and ^{240}Pu .

How does a 10% error in each of the measured isotopic abundances affect the final mass measurement?

Isotope	Error in mass measurement
^{238}Pu	<0.1%
^{242}Pu	<1.0%
^{240}Pu	~10%


Moral: ^{240}Pu is the most important isotope for accurate mass determination by a combination of neutron and gamma-ray based NDA techniques

Instructor Notes:

Neutron coincidence counting measures spontaneous fission in Plutonium. Spontaneous fission yields many isotopes important for the fuel cycle, like ^{238}Pu , ^{240}Pu , ^{242}Pu . ^{240}Pu produces most of the spontaneous fission because is present in larger quantities, therefore, it is important to obtain accurate measurements of this isotopes. A 10% error in its measurement will cause a 10% error in the total Pu mass measurement. ^{238}Pu and ^{242}Pu have higher spontaneous fission rates but are present in smaller quantities; errors in their measurements have less effect on the total accuracy of Pu mass.

Additional Information for Students:

Ratio-Based Enrichment Measurements



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Basic equation for the counts in a peak at energy (E)

$$C(E) \sim \frac{N \times BR(E) \times RE(E)}{T_{1/2}}$$

Next take ratio of two peaks from different isotopes and rearrange equation to get:

$$\frac{N^i}{N^k} = \frac{C^i(E_1)}{C^k(E_2)} \cdot \frac{T_{1/2}^i}{T_{1/2}^k} \cdot \frac{BR^k(E_2)}{BR^i(E_1)} \cdot \frac{RE(E_2)}{RE(E_1)}$$

N^i	Number of atoms of isotope i
$C^i(E_k)$	Photo-peak area at energy E_k emitted from isotope i
$T_{1/2}^i$	Half-life of isotope i
$BR^i(E_k)$	Branching ratio of gamma ray with energy E_k from isotope i
$RE(E_k)$	Relative efficiency at of gamma ray with energy E_k

The number of counts in a peak is proportional to:

- The amount of material (N)
- The activity of the material, which is proportional to $1/T_{1/2}$
- The branching ratio (BR)
- The total efficiency, which is proportional to the relative efficiency RE


If you take the ratio of the number of

counts in two peaks and rearrange, you can solve for the isotopic ratio.

Instructor Notes:

Standard expression for obtaining isotopic ratios from a gamma-ray spectrum. In this equation, the total efficiency has been rewritten in terms of the relative efficiency (RE), the geometric factors associated with the total efficiency cancel and relative efficiency includes the effects of sample self-absorption, attenuation in materials between the sample and the detector and detector efficiency. The need for only an efficiency ratio removes the problems associated with the geometric and sample reproducibility associated with absolute measurements and makes the method applicable to samples of arbitrary size, shape and composition.

Additional Information for Students:



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The Isotopic Ratio Formula

$$\frac{N^i}{N^k} = \frac{C^i(E_1)}{C^k(E_2)} \cdot \frac{T_{1/2}^i}{T_{1/2}^k} \cdot \frac{BR^k(E_2)}{BR^i(E_1)} \cdot \frac{RE(E_2)}{RE(E_1)}$$

Desired
Answer

Peak
Analysis

Nuclear
References

Nuclear
References

BR/C
Analysis

All these values are obtained from the spectrum itself or from standard nuclear references; no calibration is necessary!

How are the peak counts and relative efficiency are obtained from the spectra?

Notice that all of these values come from either the single measured spectrum or from nuclear reference data. Thus, we can find the isotopic ratio without any calibration measurements, without known geometries, and without prior detector characterization.

Instructor Notes:

Additional Information for Students:

Peak Counts and Relative Efficiency

- $C^i(E_k)$ is determined using standard spectral analysis techniques
 - $C^i(E_k)$ is a net area - proper background subtraction is critical
 - Peaks are often unresolved or overlapping; peak fitting methods are required
- A relative efficiency curve is determined using several peaks from the same isotope in the spectrum

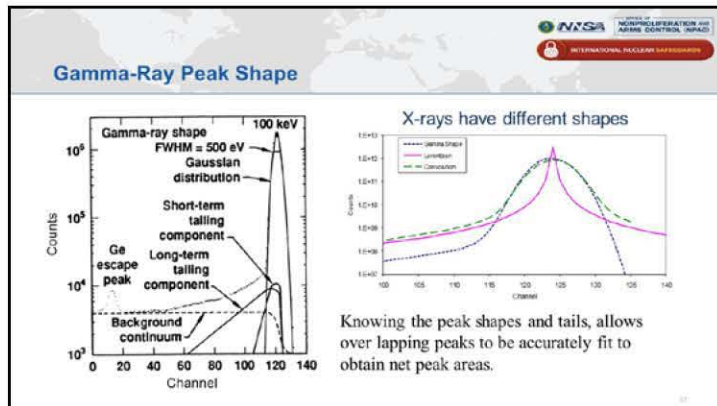


High Energy Resolution required to separate overlapping peaks!

The first physical requirement for this technique is a high resolution detector. The peak information must be extracted very accurately, which will require an HPGe detector.

Instructor Notes:

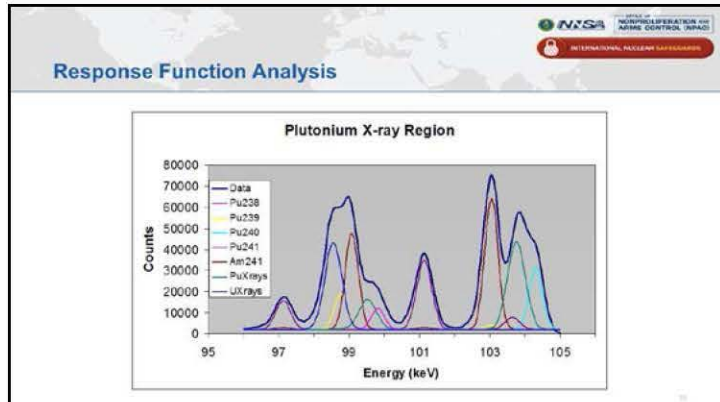
Additional Information for Students:



To accurately extract peak information, the real shape of the peaks must be known. Scattering (and poor electronic settings) can produce a low energy tailing on the peaks. Peaks from X-rays also have a fundamentally different shape, and while this is hard to manually observe even with HPGe detectors, it is a necessary consideration for peak fitting.

Instructor Notes:

Additional Information for Students:




When analyzing unshielded plutonium/uranium, the x-ray region can be used for very accurate isotopic analysis. This region contains several useful gamma-rays, as well as several x-rays. Even with HPGe detectors, the peaks in this region overlap significantly and will require response function analysis to be useful.

Instructor Notes:

The Excel sheet that produced this simulated spectrum will be available to students in the lab. You can vary the grade of the plutonium to see the effect on this region.

Additional Information for Students:

Relative Efficiency



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- To use peak area information to determine isotopic composition, the relative efficiency curve must be measured
- The efficiency curve is determined from the peaks in the spectrum
 - Requires several peaks from the same isotope for accurate determination
- Several factors influence the relative efficiency curve:
 - Detector efficiency
 - External attenuation
 - Internal attenuation
- $RE(E_k)$ is determined from the spectrum itself
- A Relative Efficiency curve can be created if you have gamma rays from the same isotope spread over a wide energy range
- If the material is isotopically homogeneous, you can combine data from multiple isotopes to make a more complete Relative Efficiency curve.


While knowing the total efficiency would be useful, it requires significantly more knowledge to calculate (detector efficiency, geometry, some material characteristics, and more). Instead, we can use the relative efficiency (RE) for this analysis, which removes many of these requirements. The RE still includes the detector efficiency, attenuation effects, etc,

but unlike the total efficiency it can be determined from the spectrum itself.

Instructor Notes:

Using relative efficiency can help simplify the analysis because it does not require ulterior measurements, it can be determined from the same spectrum being already analyzed. The relative efficiency is viewed as a function of energy. Almost any variable that perturbs the absorption or relative intensity of gamma rays emitted from the sample can affect the shape or energy dependence of the relative efficiency curve. The relative efficiency is proportional to the absolute efficiency and contains the detector efficiency, the external attenuation and the internal attenuation.

Additional Information for Students:



Relative Efficiency Equations

- Definition

$$\text{Efficiency} = \frac{\gamma\text{-rays detected}}{\gamma\text{-rays emitted in item}}$$
- General expression for peak area count rate

$$C = N \cdot \lambda \cdot BR \cdot \varepsilon$$

C = count rate in peak
 N = number of radioactive atoms
 λ = radioactive decay constant
 $\quad = \ln(2)/T_{1/2}$
 BR = branching ratio
 ε = efficiency
- Rearrange:


$$\varepsilon = \frac{1}{N \cdot \lambda} \cdot \frac{C}{BR}$$
- Relative efficiency is proportional to count rate/branching ratio

$$\varepsilon \propto \frac{C}{BR}$$


Thus, if we measure C for several peaks and divide each by their respective branching ratios, we obtain a measure of the relative efficiency.

Instructor Notes:

Additional Information for Students:



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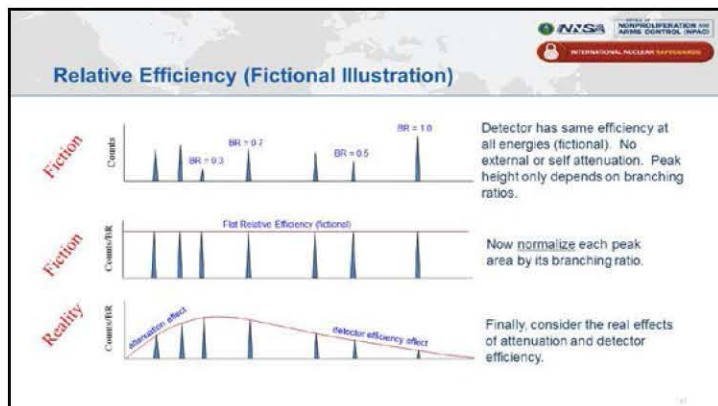
Determination of Relative Efficiency Curve

- The Relative Efficiency Curve is determined by
 - Detector absolute efficiency over the energy range
 - The effect of U/Pu self absorption
 - The effect of container shielding
 - Detector filtering
- Since the formula we are using corrects for the Relative Efficiency curve, none of these factors will affect our results
- Ratio-based programs calculate the BR/C ratios and fit that data to a Relative Efficiency Curve model

Instructor Notes:

The relative efficiency is determined from each measured spectrum by considering the energy variation of the quotient of BR/C for a series of gamma rays from a single isotope and then fitted to a relative efficiency curve model. The ratio takes into account the variations caused by the detector efficiency, the sample self-absorption and geometry, the container shielding and filters placed between the sample and the detector.

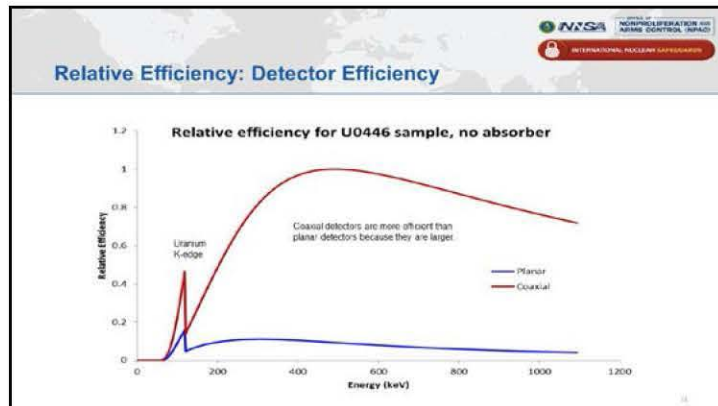
Additional Information for Students:



Consider this fictional example. In the top figure, a set of peaks is shown. If we divide the area of each peak by its branching ratio, we get the relative efficiency. If we did not have a uniformly efficient detector, we would likely observe an efficiency curve like in the last example.

Instructor Notes:

Additional Information for Students:

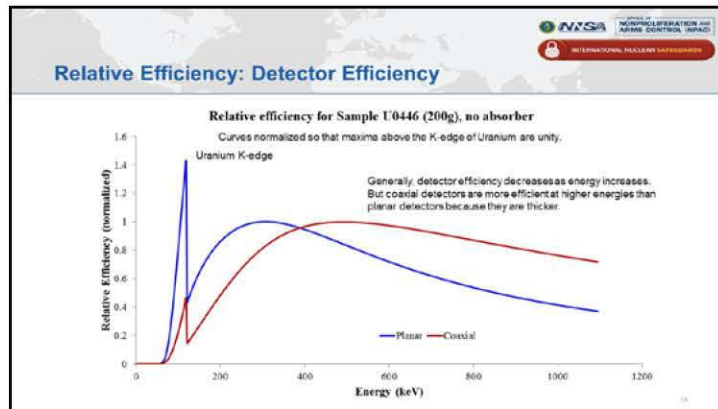


As mentioned, there are several components of the relative efficiency curve. First, it includes the detector's efficiency. Here, the coaxial detector is more efficient than the planar detector, particularly at higher energies.

Instructor Notes:

Question for the students: Why is the coaxial detector's efficiency so much higher?

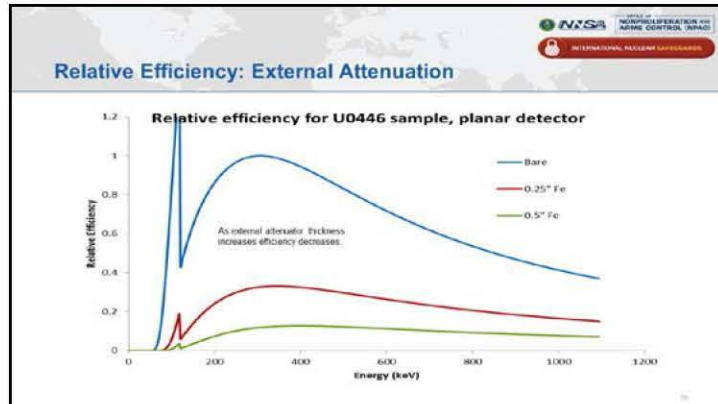
Additional Information for Students:



If we normalize each of the curves, we can better observe the relative difference between the two detectors.

Instructor Notes:

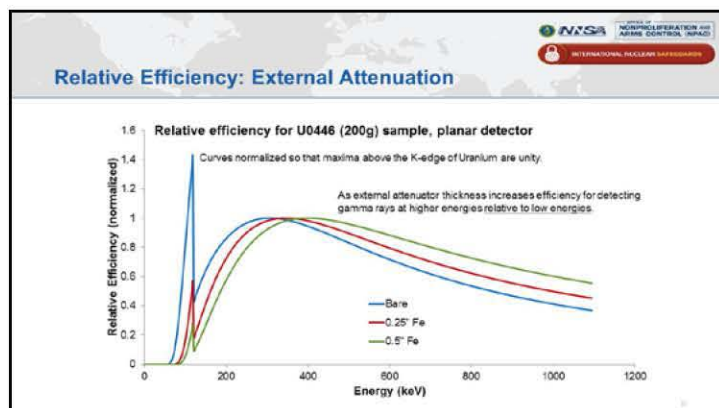
Additional Information for Students:



The next component of the relative efficiency curve is external attenuation. Shielding materials (lead bricks, steel containers, etc) have an energy-dependent shielding effect: low energy gamma-rays are much more likely to interact with them.

Instructor Notes:

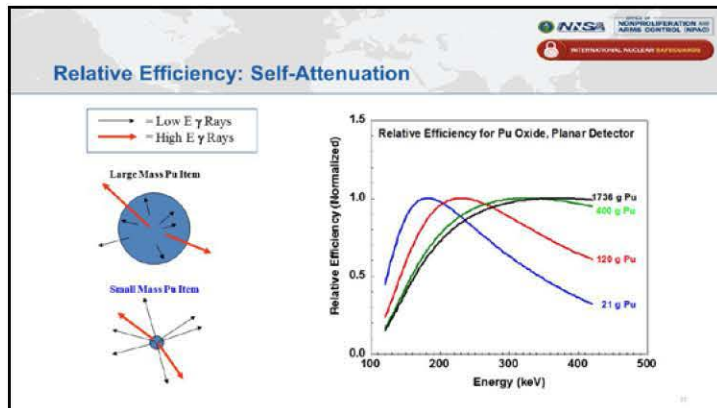
Additional Information for Students:



Comparing the normalized curves, it is clear that the effect of shielding is less at higher energies, and that shielded spectra may require using higher energy peaks for the analysis.

Instructor Notes:

Additional Information for Students:



Finally, the source itself can attenuate any emitted gamma-rays. For very small items, this is negligible; for larger items, this effect becomes much more significant.

Instructor Notes:

The figure shows the overall effect seen as the sample gets larger, which usually also means thicker. The curves are normalized to unity at their maximum value. The mean free path increases as energy increases, therefore, an isotopic measurement will see farther into the sample and hence more sample volume and mass at high energy than at low energy.

Additional Information for Students:

Use of Relative Efficiency Curve

- The fitted curve is used to calculate the RE values for the gamma-rays used to calculate the isotopic ratios
- A good Relative Efficiency curve fit is essential to the Ratio-Based measurement technique
- The data and the models can be used to draw conclusions about the measurement configuration
 - U/Pu density
 - Absorber thicknesses



All of these effects combine to give an overall relative efficiency.

Instructor Notes:

Note that we didn't discuss geometric efficiency. Why?

Additional Information for Students:

Calculating Isotopic Composition


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- The isotopic ratios are calculated from the spectrum:

$$\frac{^{235}\text{U}}{^{238}\text{U}} \quad \frac{^{234}\text{U}}{^{238}\text{U}}$$

- And we know that:

$$^{234}\text{U} + ^{235}\text{U} + ^{238}\text{U} = 1$$


- So:


$$\frac{^{234}\text{U}}{^{238}\text{U}} + \frac{^{235}\text{U}}{^{238}\text{U}} + \frac{^{238}\text{U}}{^{238}\text{U}} = \frac{1}{^{238}\text{U}}$$

The result of this analysis is a set of isotopic ratios. We can convert this to isotopic fractions with a little arithmetic.

Instructor Notes:

Additional Information for Students:





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Calculating Isotopic Composition


- We can solve $\frac{^{234}\text{U}}{^{238}\text{U}} + \frac{^{235}\text{U}}{^{238}\text{U}} + \frac{^{238}\text{U}}{^{238}\text{U}} = \frac{1}{^{238}\text{U}}$ for ^{238}U
- Which allows us to calculate ^{234}U and ^{235}U from the original ratios:


$$\frac{^{234}\text{U}}{^{238}\text{U}} \cdot ^{238}\text{U} = ^{234}\text{U}$$

$$\frac{^{235}\text{U}}{^{238}\text{U}} \cdot ^{238}\text{U} = ^{235}\text{U}$$


Instructor Notes:

Additional Information for Students:





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Calculating Isotopic Composition

- The last step is to account for ^{236}U
- ^{236}U is not measurable with gamma spectroscopy
- The operator declaration or estimate the ^{236}U content by correlation to other isotopes is used
- The other isotope fractions must be reduced (re-normalized) so all the U isotope fractions still add up to 100%


- The same process can be done for plutonium.
- For plutonium there are 5 isotopes
- ^{242}Pu is not measurable with gamma spectroscopy, same procedure as ^{236}U .

Instructor Notes:

Additional Information for Students:



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Isotopic Composition Software

- FRAM: Fixed-energy Response-function Analysis with Multiple efficiencies
- MGA: Multi Group Analysis
- Self-calibration using several gamma-ray peaks
- Peak ratio-based analysis for isotopic abundances
- Used for both uranium and plutonium
- The key difference between FRAM and other isotopic codes (such as the Multi-Group Analysis (MGA) software from LLNL) is the use of user-editable analysis parameters in FRAM

MGA and MGAU are similar software packages from LLNL; MGA analyzes plutonium, while MGAU analyzes uranium.

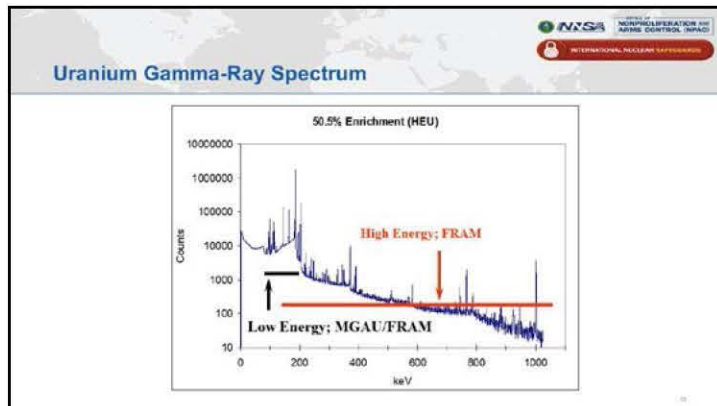
Instructor Notes:

There are multiple software able to measure isotopic composition.

FRAM: analyzes heterogeneous samples by creating two different efficiency curves, for the plutonium and the other material.

MGA is a widely used computer code for the analysis of high-resolution gamma ray spectra in order to extract the relative isotopic composition of plutonium for a diversity of items with minimal prior information.

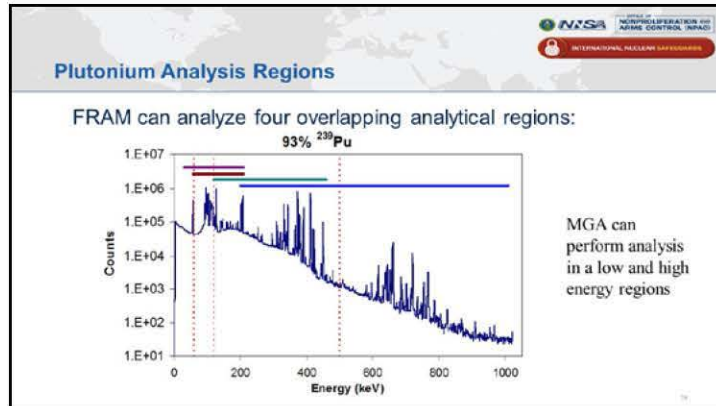
Additional Information for Students:



FRAM can use the low energy or the high energy region for the analysis. Older versions of MGAU are limited to low energy analysis.

Instructor Notes:

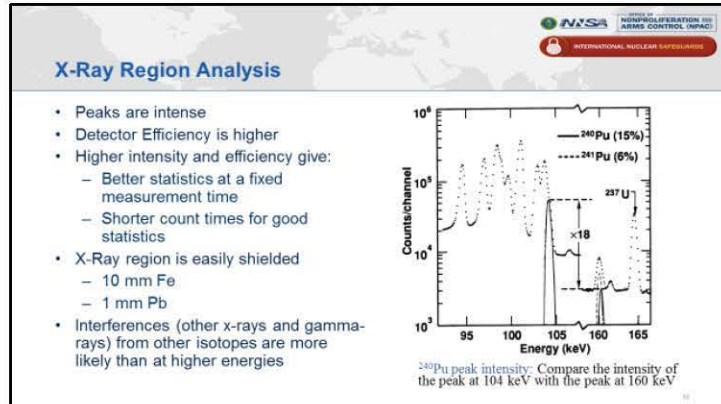
Additional Information for Students:



For plutonium, there are several different analysis regions that can be used. The detector and shielding configuration will dictate which region yields the best results.

Instructor Notes:

Additional Information for Students:



The x-ray region is (for unshielded items) typically the most intense region. It also has peaks for all of the isotopes of interest (aside from U-236 / Pu-242); this helps make the relative efficiency curve more reliable and simplifies the ratio analysis. However, this region can be easily shielded.

Instructor Notes:

Additional Information for Students:

Measure Isotope Ratios

- No Calibration Necessary
- Using **relative** efficiency and isotope **ratios** makes analysis independent of:
 - Item characteristics (size, shape, etc – no calibration required)
 - Data acquisition limitations (pulse pile up, dead time, etc)
- Analysis still requires:
 - Isotopic homogeneity
 - Pu and Am must have uniform spatial distribution

Isotopic Homogeneity:

Metal
80% Pu239
15% Pu240
Powder
80% Pu239
15% Pu240

Isotopic Heterogeneity:

Powder
80% Pu239
15% Pu240
Powder
50% Pu239
45% Pu240

The Exception:
Pu-metal in Am241 salt

- Does not work for plutonium

For uranium, the infinite thickness method was a convenient way to measure the isotopes, but it


- Requires calibration
- Requires some knowledge of configuration
- Has geometry requirements
- Requires isotopic homogeneity

Of these flaws, the isotopic ratio method only shares the homogeneity requirement.

Instructor Notes:

Additional Information for Students:

Problems to Avoid when Using the Ratio Method



- Resolution is critical in the x-ray region
 - Keep the dead time below 50%
 - Maintain a good pole-zero
- Protect the detector from other radioactive materials
 - Shielding (collimator) from other sources
 - Distance (several meters at the least) from other sources
- Ensure the item is
 - Homogenous (same isotopic composition throughout)
 - Aged for 6 months for uranium (or proper separation date is entered in FRAM)

Fresh uranium can be analyzed with MGAU, but it will require a manual correction.

Instructor Notes:

Additional Information for Students:



Instructor Notes: